

# PATENT SPECIFICATION

NO DRAWINGS

Inventors: LIONEL JAMES PARSONS and KENNETH MELDRUM

916,439



Date of filing Complete Specification Oct. 31, 1961.

Application Date Nov. 1, 1960.

No 37454/60.

Complete Specification Published Jan. 23, 1963.

Index at acceptance: —Class 15(2), B2L(1:5A:5D).

International Classification: —D06p.

## COMPLETE SPECIFICATION

### Improvements relating to the Dyeing of Acrylic Fibres

We, COURTAULDS LIMITED, a British Company, of 16, St. Martin's-le-Grand, in the City of London, England, do hereby declare the invention, for which we pray that a patent 5 may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement: —

10 This invention relates to the dyeing of acrylic fibres which term is used in this specification to include homopolymers of acrylonitrile and copolymers of acrylonitrile which contain at least 40 per cent by weight of acrylonitrile. The term therefore includes 15 the types of acrylic fibres which are available commercially containing at least 90 per cent of acrylonitrile, the acetone-soluble type of acrylonitrile-vinylidene chloride copolymers which are disclosed in British Patent Specification No. 643,198 and the specifications of our 20 Patents Nos. 849,029 and 897,697 as well as blends of polymers or copolymers at least one of which contains acrylonitrile units.

25 One of the problems associated with acrylic fibres is that of dyeing the fibres satisfactorily. Numerous proposals for improving the dyeing characteristics have been made including the copolymerisation of acrylonitrile with unsaturated compounds such as vinyl pyridine, vinyl pyrrolidone or an unsaturated acid such as itaconic acid or an allyl sulphonic acid, and the use of certain dyeing assistants in the dyebath.

30 35 We have now found that water-soluble phenols, and in particular phenol itself, are very effective dyeing assistants for dyeing acrylic fibres with disperse dyes and basic dyes. According to the present invention comprises a process for dyeing acrylic fibres as defined above in a dyebath containing a disperse dye or a basic dye and also containing a water-soluble phenol.

40 The term "water-soluble phenol" is used

to indicate a phenol which is itself soluble in water to an extent of at least 1 per cent in the dye bath under the actual dyeing conditions and does not include those phenols which are only soluble to this extent in the form of their sodium or other alkali metal salts. Ortho- and para-phenyl phenols have been found to be insufficiently soluble in water to have any beneficial effect. In addition to phenol itself, other phenols which may be used are ortho, meta- and para-cresol, thymol, resorcinol, tertiary butyl phenol, ortho-sec butyl phenol and para-isopropyl phenol. Phenols containing other substituent groups may be used to improve the dyeing; for example salicylic acid and para-chloro-phenol can be used but are not so effective as phenol and the other phenols specified above.

45 50 55 60 65 70 75 80 85 The preferred concentrations of phenol in the dyebath are from 20 to 40 grams per litre of dyebath but care should be taken to select a concentration such that the acrylic fibre is not adversely affected by the phenol under the dyeing conditions. At such concentrations the phenol is effective at any liquor:yarn ratio. We have used liquor:yarn ratios which are between 5:1 and 500:1 successfully.

The phenol is preferably added to the dyebath rather than applied separately to the material to be dyed before the dyeing operation.

75 After dyeing the acrylic fibres according to this invention, it is highly desirable to remove all or substantially all the phenol from the fibres. This may generally be accomplished by prolonged washing of the fibres with cold or warm water or by washing the fibres with hot water, for example with water at 95° C. for one minute.

The present invention may be applied to acrylic fibres in any form, for example yarn as in the hank, staple fibre, woven, knitted and

tufted fabrics and continuous filamentary tow.

The dyeing procedure used in this invention may be any of the standard procedures, for example jig-dyeing and pad-dyeing of fabrics, pack dyeing of staple fibres or in continuous dyeing processes. Spent dyebaths containing the phenol may be re-used after adjustment of the concentration.

The use of phenols as dyeing assistants in accordance with this invention enables certain acrylic fibres to be dyed effectively either for shorter times or at lower temperatures; the former is a particularly useful advantage in continuous dyeing and the latter offers advantages in the dyeing of pile fabrics or in the dyeing of fibres subject to delustring. For other types of fibre it enables deeper shades to be obtained with standard conditions. In all cases the light- and wash-fastness of the dyeings is similar to that of normal dyeings.

The invention is illustrated by the following examples, in which percentages are by weight.

EXAMPLE 1.

A yarn composed of a ternary copolymer of 93 per cent of acrylonitrile, 6 per cent of methyl acrylate and 1 per cent of itaconic acid was dyed for two hours in a bath at 80° C. containing 2 per cent of Astrazone Blue 3 RL with 20 grams per litre of phenol and 0.5 per cent (calculated on the yarn weight) of both acetic acid and sodium acetate; the liquor to yarn ratio was 40:1. The yarn was then washed off and dried.

The yarn obtained was dyed to a deep blue or purple shade whereas a yarn dyed under similar conditions in the absence of phenol was coloured light blue. The yarn was found on analysis to contain no trace of phenol and had satisfactory wash- and light-fastness.

EXAMPLE 2.

A yarn composed of 48 per cent of acrylonitrile and 52 per cent of vinylidene chloride produced by dry spinning an acetone solution of the copolymer was dyed for two hours at 98° C. using the dye bath and liquor:yarn ratio as described in Example 1. Similar results were obtained to those described in Example 1.

EXAMPLE 3.

A fabric prepared from the yarn specified in Example 1 was dyed under the conditions in Example 1 with the only variations being (a) the use of 2 per cent of the dye Sevron Red L in place of the 2 per cent of Astrazone Blue 3 RL and (b) the use of 20 grams per litre of para-cresol in place of the phenol. The fabric obtained was dyed deep red whereas in the absence of the para-cresol the fabric is only dyed to a pale pink.

EXAMPLE 4.

15 denier, 2½ inch staple fibre of the same

composition as the yarn in Example 1 was dyed in bulk for 35 seconds at 90° C. in a dyebath containing in solution 0.1 per cent of Sevron Brilliant Red 4 G and 40 grams/litre of phenol. The fibre was then mangled, rinsed for one minute with water at 95° C. and dried. It was dyed to a deep red whereas in the absence of the phenol the fibre is only dyed to a pale pink.

EXAMPLE 5.

A yarn similar to that described in Example 2 was dyed for two hours at 98° C. in a bath containing 1 per cent of Serilic Dark Blue GR and 20 grams/litre of phenol. After washing and drying a medium blue yarn was obtained. In the absence of the phenol the shade obtained is pale blue.

EXAMPLE 6.

Yarn composed of the vinylchloride-acrylonitrile copolymer sold under the Registered Trade Mark "Dynel" was dyed for 1 hour at 70° C. in a bath containing 2 per cent Duranol Brilliant Blue BN and 20 grams/litre of phenol. After washing and drying a deep blue yarn was obtained; in the absence of the phenol the shade obtained is light blue.

EXAMPLE 7.

A tufted candlewick fabric with a pile composed of a yarn as specified in Example 1 and a cotton backing was dyed in a rotary washing machine at 50° C. for 1 hour using a dye liquor containing 0.1 per cent of Cibacet Sapphire Blue G 300 and 40 grams/litre of phenol; the liquor:yarn ratio was 15:1. The fabric was then washed off and dried. The resultant fabric was dyed medium blue and the pile was substantially unaffected. In the absence of phenol the above conditions gave no dyeing at all; furthermore if the temperature of the dyebath is raised, also in the absence of phenol, so as to achieve a similar colour, the pile is flattened.

EXAMPLE 8.

The bright acrylic staple fibre sold under the Registered Trade Mark "Verel" was dyed for 2 hours at 40° C. in a bath containing 1 per cent of Maxilon Red BL, 20 grams/litre of phenol and 0.5 per cent, calculated on the weight of yarn, of both acetic acid and sodium acetate. After washing off and drying, a medium red yarn was obtained in which the lustre was preserved. Under the dyeing conditions specified but without phenol only a slight pink shade is obtained. If the temperature is raised to 70° C. so as to get the same amount of dye on the fibre, the fibre obtained is delustrated.

EXAMPLE 9.

A 4½ denier per filament tow of filaments having the composition set out in Example

1 was padded at 40° C. through a solution containing per litre, 20 grams of Astrazone Red GTL, 40 grams of phenol, 40 grams of urea, 5 grams of an emulsifying agent and

5 1 gram of acetic acid. The tow was immediately steamed for 5 minutes in a festoon steamer, washed for 2 minutes in a solution containing 1 gram/litre of a non-ionic detergent (AL30 sold by Imperial Chemical Industries Ltd.) and dried. The tow was dyed to a dark red shade and the filaments were substantially fully penetrated.

10 WHAT WE CLAIM IS:—

15 1. A process for dyeing acrylic fibres as hereinbefore defined wherein the fibres are dyed in a dyebath containing a disperse dye or a basic dye and also containing a water-

soluble phenol as hereinbefore defined.

2. A process as claimed in Claim 1 wherein the dyebath contains from 20 to 40 grams per litre of the water-soluble phenol. 20

3. A process as claimed in Claim 1 or 2 wherein the water-soluble phenol used is phenol itself. 25

4. A process as claimed in Claim 1 carried out substantially as described in any of the foregoing examples.

5. Acrylic fibres which have been dyed by the process claimed in any of the preceding claims. 30

J. Y. & G. W. JOHNSON,  
47, Lincoln's Inn Fields, London, W.C.2,  
Chartered Patent Agents,  
Agents for the Applicants.

Leamington Spa: Printed for Her Majesty's Stationery Office by the Courier Press.—1963.  
Published at The Patent Office, 25, Southampton Buildings, London, W.C.2, from which copies may be obtained.